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The interaction of dislocations with grain boundaries in polycrystalline ice have been studied using *in situ* deformation sychrotron x-ray topography. Specimen preparation was performed in the Ice Research Laboratory at Dartmouth College, and x-ray topography was performed at the National Sychrotron Light Source at Brookhaven National Laboratory, in collaboration with Prof. M. Dudley (SUNY at Stony Brook) and Dr. D. Black (NIST).

We have demonstrated that x-radiation can be used to study ice without affecting the dislocations present. The main part of the work has demonstrated that normally basal dislocations are emitted from grain boundary facets during grain boundary sliding. However, in situations where basal slip is suppressed, by having no resolved shear stresses on the basal plane, non-basal slip occurs. Normally, dislocations are emitted into both adjacent grains from a grain boundary facet, glide across the grain and pile-up at the opposite boundary. However, it has been demonstrated that these pile-ups can lead to slip transmission for certain geometrical arrangements of the basal planes, on either side of the grain boundary..

The project also briefly explored dislocation behavior ahead of loaded notches in single crystals, and the effect of high concentrations of HCl on the velocity of dislocations.

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X-RAY TOPOGRAPHIC STUDIES OF DISLOCATION/GRAIN BOUNDARY INTERACTIONS IN ICE

Final Report

Ian Baker

October 18, 1996

U.S. ARMY RESEARCH OFFICE

Contract no. DAAH04-93-G-0061

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INTRODUCTION

Work funded by ARO (Grant No. DAAH04-93-G-0061) was initiated on March 1st, 1993, under the direction of the author on "X-ray Topographic Studies of Dislocation/Grain Boundary Interactions in Ice". The aim of the project which was to examine the effect of geometry, temperature and stress on the dislocation/grain boundary interactions in ice. For this purpose, bicrystalline and polycrystalline specimens of high-purity, bubble-free, freshwater ice were prepared in the Ice Research Laboratory at Dartmouth College and stress-controlled *in-situ* deformation experiments were performed on them using white radiation on X-ray beamline X-19C on the synchrotron at the National Synchrotron Light Source, Brookhaven National Laboratory, Long Island, NY. The *in-situ* compression experiments were performed for a variety of geometrical arrangements and at different testing temperatures. In addition, the dislocation activity around notches in several ice single crystals of different orientations were studied whilst under load using white radiation.

In contrast, the effect of high concentrations of HCl on the velocity of dislocations in ice was studied in single crystals using monochromatic radiation. In order to validate the use of x-radiation in studying dislocations in ice, a number of experiments were also performed using monochromatic x-radiation to determine if it affected the creep behavior. This part of the work was performed on X-ray beamline X-23A.

THE EFFECT OF X-RADIATION ON THE DEFORMATION OF SINGLE CRYSTAL ICE

Several ice single crystals under were tensile-tested (up to 0.15 W/m^2) and without x-radiation in a jig that was originally designed and built for compression experiments. All specimens were deformed under a constant load of 20 ± 0.3N at a temperature of -13 ± 0.1°C. The load and temperature were so chosen that ice can be deformed appreciably within an

appropriate time, which was predicted based on the experimental data of previous workers on the creep deformation of single crystal ice[1].

One point worth noting is that the crystals used were very similar, i.e., both the orientations and initial dislocation densities were very similar. To exclude effects due to subgrain boundaries, only specimens with no observed subgrain boundaries were used for the tensile tests. This was ensured by making the specimens from the same crystal and cutting them in the same orientation.

These direct measurements of the creep behavior of single crystals of ice with and without X-ray radiation showed that X-ray radiation at the intensity used for x-ray topography does not have any significant effect on the plastic deformation of ice.

DISLOCATION/GRAIN BOUNDARY INTERACTIONS

In ice single crystals, dislocations have been observed to nucleate and multiply via a variety of mechanisms, i.e. Frank-Read sources, pole mechanisms and surface nucleation mechanisms[2] Thus, the question was asked: what role do grain boundaries, which are present in all naturally-occurring ice, play in the nucleation of dislocations? Synchrotron x-ray topography of bubble-free, high-purity freshwater polycrystalline ice showed that:-

1. dislocation nucleation occurs mainly at facets on the grain boundaries, slip occurring primarily by glide of semi-hexagonal dislocation loops on the basal plane.

2. for a grain oriented so that the resolved shear stress on the basal plane (from the far-field stress) is zero, then non-basal slip can occur, with dislocation nucleation at the grain boundaries.

3. slip transmission can occur through grain boundaries in ice, but only if the grains involved are in a special orientation relationship with one another.

4. dislocation pile-ups form at grain boundaries during loading and relax during unloading/annealing.

These observations are aiding the modeling of the mechanical properties of polycrystalline ice by Prof.s E.M. Schulson and H. J. Frost at Dartmouth and Dr. D. Cole at CRREL.

THE EFFECT OF IMPURITIES ON THE DEFORMATION OF SINGLE CRYSTAL ICE

Dislocation motion was studied in HCl-doped single crystal ice using monochromatic radiation. For lightly-doped (1x 10^{-6} M) specimens, the HCl was diffused in from the surface after the crystal was dipped in HCl solution. More highly-doped (1.9 x 10^{-4} M) specimens were grown directly from water containing 1x 10^{-3} M of HCl. The dislocation velocity was determined by imaging the dislocations at successively increasing loads and dividing by the time between the increased loads. The experiments were performed over a range of temperatures.

It was shown that a concentration of ~1 x 10^{-6} M of HCl does not produce any change in dislocation mobility in ice. This may be because the concentration of intrinsic point defects swamp that of the extrinsic defects introduced by the HCl. In contrast, the velocity of dislocations in heavily HCl-doped (1 x 10^{-4} M) ice was at least 2.6 times larger than that previously measured in pure ice. The latter observation suggests that the softening effect produced by HCl-doping(3) might be due to the higher bond reorientation rate at the dislocation core. However, at this point, an effect due to the increase in mobile dislocation density from the HCl-doping cannot be ruled out as a cause of the softening effect since all the HCl-doped specimens examined contained higher dislocation densities than undoped ice.

DEFORMATION OF NOTCHED ICE SINGLE CRYSTALS

Cracks and notches are very important in the deformation of ice. Cracking in ice has been much studied recently on a macroscopic level at Dartmouth through ARO support of Prof.s E.M. Schulson, H. J. Frost and V. Gupta. However, what is happening at the dislocation level is unknown. The objective of this part of the project was to use x-ray topography to study the deformation of notched ice. The three specimen orientations studied in this work are shown in Figure 1. Eventually, it is hoped that polycrystals will be studied at a variety of temperatures and strain rates but, initially, single crystals were examined so that the observations can be matched to calculations of the stress fields. A key initial question was whether specimens could be made with a sufficiently low level of damage. Preliminary tensile tests performed on single crystal ice showed that notched ice could be produced with minimal damage to the specimen and that both the notch-tip strain field and the associated dislocation movement could be observed.

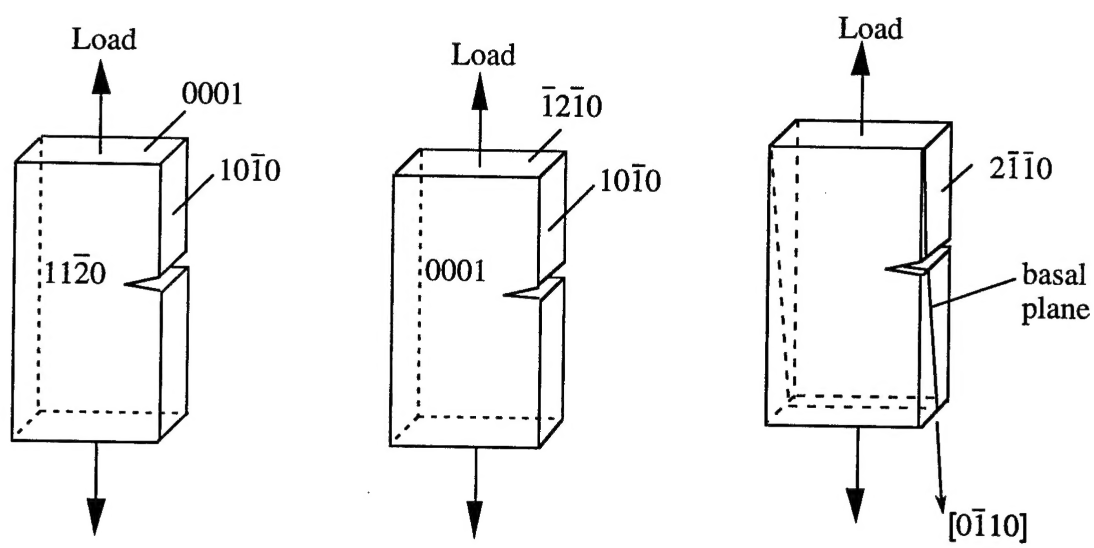

Figure 1. Single crystal orientations using to study dislocation activity ahead of loaded notches.

It was found that the plastic deformation of notched ice single crystals was mainly caused by the movement of the dominant dislocations which lie on the basal plane. It was also observed

that short segments of dislocations on the non-basal plane play an important role in the generation of screw dislocations on the basal plane. It was shown that the dislocation/crack tip interaction mechanism depends strongly on the crystal orientation relative to the loading direction and the crack plane, i.e. the slip system with the highest Schmid factor is the most active in single crystal ice.

Importantly, no dislocation-free zone was found in any of the specimens, and it was concluded that the commonly-used etch pit and replication method has limited utility for observing the motion of dislocations in ice crystals.

SCIENTIFIC PERSONNEL SUPPORTED

In addition to the author, several others were involved during the course of this project and fully or partially supported by ARO funds :

D. Cullen - Ph.D. student

X. Hu - M.S. student

K. Jia - M.S. student

F. Liu - Post-Doctoral Fellow

Several undergraduates were also employed during the course of the project.

FUTURE WORK

In the future, the research program will use *in-situ* synchrotron x-ray topography of both single crystal and polycrystalline ice to study both cracks and notches in ice, and to determine dislocations structures in mechanically-tested bulk specimens. The goal will be to provide fundamental information on dislocation behavior that will enable the modeling of the deformation of ice to move from the phenomenological to the micromechanisms based.

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